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Use of Dialdehyde Starch in Coating Color Formulation

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USE OF DIALDEHYDE STARCH
IN
COATING COLOR FORMULATION /

Senior Student Thesis

Produced in Partial Fulfillment of the
Requirements for the Degree of Bachelor
of Science in Paper Technology

By

Craig F. Panse

//

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June, 1960

USE OF DIALDEHYDE STARCH IN COATING COLOR FORMULATION

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USE OF DIALDEHYDE STARCH IN COATING COLOR FORMULATION

ABSTRACT

A literature survey was conducted to determine what work, if any, had been done on dialdehyde starch as a coating adhesive. As a result of the survey, it was decided that experimental work would be started with the hope of overcoming some of the difficulties which confronted previous investigators and investigating further the use of dialdehyde starch as an adhesive.

After experimental preparation and evaluation of the coated and aftertreated sheets, the following conclusions were formed:

1. The addition of ten percent borax buffered or bisulphite buffered dialdehyde starch to conventional oxidized starch increases the surface strength of the coating as well as the wet rub resistance.
2. Aftertreatment with the starch dispersions increases the surface strength of the coating and the wet rub resistance.
3. Bisulphite buffered dialdehyde starch is a better coating adhesive and "waterproofer" than borax buffered dialdehyde starch.

USE OF DIALDEHYDE STARCH IN COATING COLOR FORMULATION

LITERATURE SURVEY

According to Thompson (1) "paper coating colors consist essentially of an aqueous dispersion of pigments and adhesives, together with various auxiliary agents which modify the rheological properties of the color as well as the characteristics of the applied coating."

Of all the common types of adhesives used today, such as casein, soy protein, starch, and synthetic resins, only one particular group of starches will be considered for experimental work, namely oxidized starches; in particular the use of a starch produced by oxidation with periodic acid will be explored.

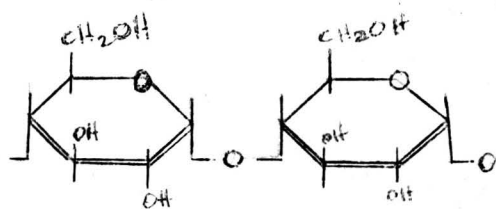
Starch must be modified before it can be used as a coating adhesive because a few percent by weight of native starch when cooked in water will form a thick, heavy dispersion. The application of this starch as a coating color is made difficult by the high viscosity, despite its good adhesive quality. Normally, a decrease in viscosity reduces adhesiveness. This fact has a definite effect in starch modification, since the function of modification is to produce a starch which has sufficient adhesive strength but yet has flow characteristics which permit its use in a coating color (2).

In each of the methods of modifying starches, the intent is to reduce the viscosity and improve the dispersion stability of the system. The character of the modified starch produced may be varied by the methods employed and the conditions

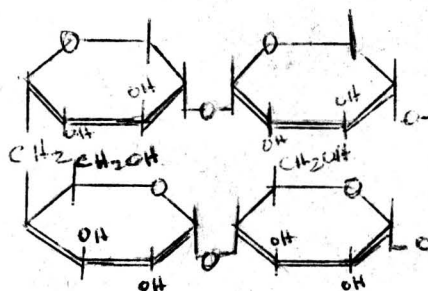
of the method. Modified starches are usually graded by the viscosity of their dispersion.

CHEMICAL COMPOSITION OF NATIVE STARCH

Starch is a globular hydrophilic carbohydrate. It has the same empirical formula as cellulose; however, the glucose units in the starch linear chains are connected by alpha linkages (2). Two species of starch molecules make up the starch granule, the straight chain (amylose) and the branched chain (amylopectin.)



Amylose



Amylopectin

It is generally accepted that the amylose and amylopectin chains are linked by alpha linkages at the 1,4 carbons. However, the branches of the amylopectin are bound to the major chains through 1,6 linkages. Amylose has higher adhesive strength, greater tendency to gel, greater viscosity, and lower dispersion stability than amylopectin. In the modification of starch, the reduction of viscosity and improvement in stability is normally brought about by compromising the adhesive and viscosity properties by selective attack on the straight chain portion.

MODIFIED STARCHES

The following modified starches will be discussed: dex-
trins, acid modified starches, enzyme converted starches, ox-
idized starches and other chemically modified starches.

DEXTRINS. "The term dextrin has been loosely applied to
certain starch degradation products made from common starch
by the action of enzyme, heat, acid or other agents, or by any
combination of these agents" (3); however, the term dextrin
usually refers to the heat treatment or torrefaction of dry
starch at high temperatures (250 to 400 degrees Fahrenheit)
for various periods of time. The products of this method
have increased solubility, decreased viscosity, increased
reducing power, altered color and changed adhesive character.
The term British Gums refers to the dextrans produced by
roasting neutral or slightly alkaline starches at the higher
temperatures.

Small amounts of acid, usually hydrochloric acid, added
to the starch, catalyze the reaction producing the canary
dextrans with lighter colors, higher solubilities, and much
lower viscosities than British Gum. A canary-yellow color
characterizes these products.

The white dextrans are produced by using larger quanti-
ties of acid as a catalyst (up to 0.12 percent hydrochloric
acid) and roasting at much lower temperatures. The white
dextrans are characterized by the variation in water solu-
bility (from 5 percent to 90 percent), their low to high vis-
cosity, and a pronounced starchy character, especially in the
ranges of low solubility.

The mechanism of heat modification and properties resulting therefrom have been discussed by Keeney (2).

The dextrins find application in low viscosity and high percent (60 percent - 70 percent) solids coating. The canary dextrins and British Gums are better suited to paper coating than white dextrins because of better pigment binding strength.

ACID MODIFIED STARCHES. Acid modified starches are produced by adding small amounts of either hydrochloric or sulphuric acid to a suspension of common starch in water and holding the starch in suspension at 120 to 130 degrees Fahrenheit for various periods of time. The conversion is effected on the unswollen granule, since this temperature does not gelatinize the starch. The suspension is then neutralized with soda ash, filtered, washed, and dried. Products of various viscosities can be obtained by controlling the time of reaction, the amount of catalyst and the temperature. When oxidized and acid modified starches at the same viscosity are allowed to cool from an elevated temperature, the acid modified starches give very heavy semi-solid pastes, while the starches remain fluid and workable. Acid modified starches also have less tack and adhesive character. The resulting gelling character and lack of "adhesiveness" of acid modified starches render them of little value as coating adhesives (3).

ENZYME CONVERTED STARCH. Enzyme conversion is one of the most economical methods of modifying starch. The ease of adjustment of the degree of conversion to produce considerable versatility in the viscosity levels is one of the major advantages of this process. The conversion of native starch by enzymes is carried out according to a prescribed cycle. The characteristic of the starch produced is a function of the

operating conditions which include type and amount of enzyme, cooking cycle, time and temperature (2).

OTHER CHEMICAL MODIFICATIONS OF STARCH. According to Keeney (2), "workable but relatively expensive starch adhesives are produced by other methods of starch modification, especially the formation of starch derivatives. These procedures consist of blocking the groups in the starch molecule which produce the undesirable characteristics of aggregation and high viscosity." Such groups as acetate in a low degree of substitution (one substituent (3) group for each 20 - 200 glucosyl units) produce starch acetate which was one of the earliest of the starch derivatives.

Another possible use of the starch derivatives is the introduction of bifunctional groups into the starch molecule to permit the cross-linking and subsequent waterproofing of the starch (2). The usual approach to waterproofing of starch is through formaldehyde resins of the urea or melamine type and their reaction with the starch.

Other derivatives of starch are produced by replacing the hydrogen atoms of the hydroxyl groups and by forming other groupings such as ethers, esters, acetals, formals and metallic alcoholates. Hydroxyethyl and hydroxypropyl ethers of starch are examples of this type of low substituted derivatives. The solubility and stability of the hydroxyethyl derivative makes it useful in coating application.

STARCHES OXIDIZED BY HYPOCHLORITES. Of the many different methods of oxidizing starches such as treatment with peroxides, permanganate, Activin or Chloramin-T, perborates,

persulphates, hexavalent chromium salts, and other oxidizing agents, only two will be discussed here. The two types are treatment of starch with either periodic acid, resulting in a dialdehyde starch, or with hypochlorite. The product formed by hypochlorite oxidation has been called "chlorinated starch" which is a misnomer because the chlorine does not enter into the starch molecule but rather serves to provide the oxidizing potential. (4)

The hypochlorite oxidation is brought about by the reduction of the active chlorine in sodium hypochlorite with the subsequent release of $1/2$ mole of oxygen.

According to Newton (4) oxidation of starch with hypochlorites appears to give four different types of reactions: (a) oxidation of aldehyde groups to carboxyl groups, (b) oxidation of primary alcohol groups to carboxyl groups, (c) oxidation of secondary alcohol groups to ketone groups, and (d) oxidation of glycol groups to dialdehydes and then to dicarboxylic acids. It is possible that any or all of these types of oxidation may occur simultaneously.

Newton (4) states that the exact reaction mechanism is not established. However, commercial oxidation definitely introduces carboxyl groups into the starch molecule, the finished product having some of the characteristics of an organic acid. The acid groups are neutralized by the excess caustic soda. The average oxidized starch has approximately 90 percent of the carboxyl groups neutralized with sodium ions. Newton continues that Pollock and Campbell observed considerable oxidation at the glycol group, resulting in production

of free formic acid during oxidation and in a break in the chain between the third and fourth carbon atoms. Additional experimental proof of this observation would aid in explaining some of the viscosity changes caused by oxidation.

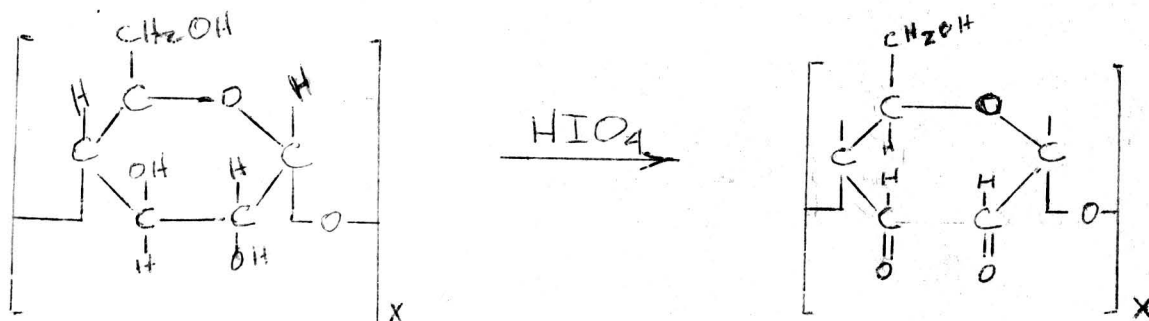
Hypochlorite oxidized starches are produced commercially by treating aqueous starch suspensions (about 35 percent dry starch) with a hypochlorite solution (6 percent to 8 percent active chlorine) containing a slight excess of sodium hydroxide. By adding the hypochlorite solution in small portions and dissipating the heat generated by means of an adequate cooling system, the temperature can be kept between 90 degrees and 125 degrees Fahrenheit.

When the desired degree of oxidation is approaching, a sample of the starch is removed, filtered, and washed. The fluidity of a paste of definite concentration prepared from the cake is determined. When a product of the desired fluidity is obtained, the oxidation is stopped by adding an anti-chlor (usually sodium bisulphite), and the slurry is adjusted to the desired pH level (usually 3 to 7), filtered and washed. After water removal to about a 47 percent to 50 percent moisture content, the oxidized starch is dried. A full range of oxidized starches is produced by varying the quantity of hypochlorite used and the time, temperature, and pH of the reaction (4,5). The oxidation of the starch results in (a) a shorter cooking time, (b) higher fluidity, (c) increased adhesiveness, (d) lower rate of congealing and (e) less turbidity of oxidized starch suspension than found for the parent starch. Films formed by drying oxidized starch are characterized by tough and horny nature in comparison with the extremely brittle property of films formed from unoxidized,

acid modified, or dextrinized starch pastes.

Oxidized starches are made over a wide viscosity range. Only the highly oxidized starches are used in coating operations. These have relatively high adhesive strength, good flow properties and excellent color. They finish and calender well and produce good coated surfaces.

DIALDEHYDE STARCHES. The selective oxidation of 1, 2 - glycols by periodate ions has been known for many years. This technique was applied to starch by Jackson and Hudson (7), who established the formula of the product resulting from this specific oxidation. The reaction proceeds according to the following scheme:



The pyranose ring is opened and a non-cyclic "polymeric dialdehyde" is formed (6).

The oxidation is carried out by suspending ungelatinized corn starch in an excess of 0.58 M aqueous periodic acid solution and allowing the reaction to proceed at room temperature. At the end of twenty-four hours, the quantity of oxidant consumed approximates closely one molecular equivalent, which is the theoretical amount required to oxidize the hexose unit of starch (I) to the corresponding dialdehyde (II). Since the consumption of periodic acid proceeds at a greatly diminished rate after the consumption of one mole of oxidiz-

ing agent, it is apparent that the principal reaction is completed at this stage.

The oxidation of starch by periodic acid gives the same results whether the oxidation is carried out on the native granules or on gelatinized starch. A detailed discussion of the mechanism of the periodate oxidation is given by Kerr (5).

Products of the dialdehyde formed by the mole-for-mole periodic acid oxidation of corn starch are as follows: the product is insoluble in cold water, soluble in hot tap water, gives no color reaction with iodine, reduces Fehling's solution, forms an amorphous precipitate with phenylhydrazine at 25 degrees Centigrade, is not attacked by malt diastase, and has a specific rotation of about +9 degrees at 20 degrees Centigrade in sodium light (5).

Aqueous solutions up to 10 percent concentration of dialdehyde starch can readily be prepared by heating the slurry 10-15 minutes at 90-100 degrees Centigrade with agitation. The dialdehydes of lower percent of oxidation are more soluble than the dialdehydes of high oxidation, but may show a pasting behavior which is more like that of ordinary starch.

The partial hydrolytic degradation of the polymer (8), causing a brown or tan color, can be avoided by preparing solutions in a buffered aqueous media. Aqueous solutions of up to 50 percent concentration can be prepared using sodium acetate as a buffer with a resulting pH of 5.0 - 5.5. Solutions of 12.5 percent concentration can be prepared with borax as buffer resulting in a pH of 7-8. Other buffers used

are sodium bicarbonate and di-sodium phosphate. Sodium bisulphite and ammonium bisulphite addition products of dialdehyde starch can be made, producing a compound which is slightly soluble in water at room temperature and in concentrations of 3-5 percent by weight forms a highly viscous solution showing pasty characteristics.

Dialdehyde starches that are known (6) react with: urea, melamine and its derivatives, phenylhydrazine and derivatives, bisulphites, acrylamide, ammonia, amino acids, hydroxylamine, amines and polyamines, alcohols, phenols, hydrocyanic acid, protein and protein fiber (animal glue, gelatin, albumins, casein, zein), polyamides, sodium borohydride, alkali (to form degradation products), and alcoholic hydrogen chloride. Other reactions of dialdehyde starch are oxidation, hydrogenolysis and hydrolysis with sulfurous acid.

USE OF DIALDEHYDE STARCH IN THE PAPER INDUSTRY

A recent article (9) discusses the application of periodate oxystarches in the paper industry as fiber deflocculating agents, beater adhesives, coating adhesives and surface-sizing agents. Although dialdehyde starches were satisfactory as tub-sizing agents, their application as beater adhesives and coating adhesives was disappointing. It was decided that experimental work would be done with the goal of overcoming some of the difficulties which confronted previous investigators.

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EXPERIMENTAL DESIGN

The objective of this project is to explore the use of dialdehyde starch in coating color formulation and its effect on the properties of the coated sheet. To carry out these objectives, the following experimental design has been planned:

Three dispersions of starch will be prepared, one of conventional oxidized starch, one of dialdehyde starch prepared with sodium bisulphite as a buffer, and one of borax buffered dialdehyde starch.

Coating colors will be prepared using twenty percent (based on pigment) conventional oxidized starch (formula I), twenty percent borax buffered dialdehyde starch (formula II), and twenty percent bisulphite buffered dialdehyde starch (formula III). Intermediate coating colors will be prepared by replacing ten percent of the oxidized starch in formula I with those starches in formula II and III. In addition to the adhesives, the color will contain water, clay, soap and preservative. The coating color will be applied with R.D. wire wound doctor rods at a coat weight of fourteen pounds per ream (25 x 38-500). Before supercalendering, some sheets from each group will be aftertreated with ten percent solutions of each of the three kinds of starch and applied with a Bird film applicator. All of the sheets will be supercalendered under controlled conditions and tested for wax pick, wet rub resistance, and gloss. Additional methods of evaluation will be used, if deemed advisable.

DESCRIPTION OF EXPERIMENTS

The materials used in the experiments were; conventional oxidized starch (Stayco M.U.), dialdehyde starch (90 percent oxidized Sumstar S), predispersed clay (Edgar H. T.), tetra sodium pyrophosphate, commercial soap flakes (Ivory soap), sodium tetra borate anhydrous, sodium bisulphite, sodium pentachlorophenate (as preservative), and raw stock obtained in the store room.

PREPARATION FOR COATING. The preliminary experiment was to find the best solids content at which the conventional starch could be cooked to yield optimum viscosity. Three dispersions were made containing 12, 18, and 24 percent solids. The dispersions were heated to 180°F. and held there for twenty minutes. After cooling to 80°-90°F. the viscosity of the dispersions was noted. The dispersions of 24 percent solids was very viscous, but satisfactory for use in a coating color.

In preparation for making the coating colors, the predispersed clay slip was made. 5000 grams of clay, 2143 milliliters of water and 15 grams of tetra sodium pyrophosphate (TSPP) were used in the final slip. The water and TSPP was put in a Day sigma blade kneader and the clay was slowly added. The seventy percent dispersion was put in a clean gallon jar and tightly closed to be used throughout the thesis. The pH of the clay slip was 7.6 when checked on the Beckman pH meter.

Ten percent solutions of sodium pentachlorophenate and Ivory soap were prepared and stored in clean jars to be used throughout the experiments.

Ten sheets of raw stock were dried in the Precision Scientific oven at 105°C. and weighed. The basis weight was calculated, averaged, and used as the raw stock sheet weight. The basis weight was 41.3 pounds per ream (25x38-500).

COATING COLOR ADJUSTMENTS The following procedure was carried out on all of the succeeding coating colors, with only slight variations which will be noted later:

1. Temperature adjusted to 80°-90°F.
2. pH adjusted to 8.5 with concentrated ammonium hydroxide.
3. Viscosity adjusted to 350 \pm 25 centipoises at 100 rpm with a number two spindle on the Brookfield viscometer.
4. Screening through a 150 mesh U.S. Standard sieve.
5. Foam removed by centrifuging for three minutes at 1500 rpm in the Universal centrifuge.
6. 14 \pm 1 pounds of coating applied by means of R.D. wire wound rods.
7. Drying for four minutes at 105° C in the Precision Scientific Company oven.

COATING SERIES I The first coating color prepared in series I contained 100 percent conventional oxidized starch as adhesive. A quantity of 417 grams of 24 percent solids starch dispersion was cooked at 200°F for thirty minutes and added to 715 grams of 70 percent clay slip. After thorough

stirring, 20 grams of soap (10 percent solids), 25 grams of preservative (10 percent solids), and 32 grams of tap water (used in all coating formulation) brought the total solids content to 50 percent.

The pH of the color was adjusted to 8.8 with concentrated ammonium hydroxide and enough water added to bring the viscosity down to 380 centipoises with a number two spindle at 100 rpm. The coating was screened and centrifuged and the hard scum, formed on the surface of the coating in the centrifuge tube, was removed and discarded. The solids content was determined to be 46.2 percent.

The raw stock was coated by placing it on a glass plate and under the edge of a plastic apron. A bead of the coating color was poured onto the apron and drawn down from the apron across the sheets. The use of the apron reduced the change in solids content by absorption of water into the sheet. By using different rods the correct coat weight of fourteen pounds per ream (25x38-500) could be obtained. This coating color was applied with a number twelve rod. The coated sheets were dried in the oven at 105°C. for four minutes, trimmed to six by eight inches, put back in the oven again for several seconds and quickly weighed on a Hermann Paper Company sheet balance. The weight obtained was multiplied by 21.8 to get the basis weight. To obtain the coat weight the raw stock basis weight, namely 41.3 pounds, was subtracted from the weight of the dried coated sheet. Several of these sheets were saved for aftertreatment.

The second coating color prepared in series I contained: 715 grams of 70 percent solids clay slip, 100 grams of 10 percent solids dialdehyde dispersion wax buffer, 360 grams of 25 percent solids conventional oxidized starch dispersion, 20 grams of 10 percent solids soap solution, and 25 grams of 10 percent solids preservative solution. The preparation of the borax buffered dialdehyde starch is described in the section on aftertreatments. The adhesive contained 90 percent conventional oxidized starch and 10 percent dialdehyde starch.

The conventional oxidized starch dispersion and additive were blended into the clay slip with the dialdehyde starch being added last. The pH of the color was adjusted to 8.9 and enough water added to bring the viscosity to 376 centipoises at 100 rpm with a number two spindle. The total solids content of this coating color was 47.2 percent.

After coating with a number 12 wire wound rod and drying, several sheets were saved for aftertreatment with conventional oxidized starch and borax buffered dialdehyde starch.

SERIES I AFTERTREATMENT Several sheets were after-treated with ten percent dispersions of conventional oxidized starch, borax buffered dialdehyde starch, or bisulphite buffered dialdehyde starch to determine the effect of this additional process. The ten percent solutions were applied with a 0.001 inch Bird film applicator by drawing a bead of the dispersion off the plastic apron across the coated sheet.

There was no significant increase in basis weight as a result of aftertreatment.

The 10 percent dispersion of conventional oxidized starch was cooked for thirty minutes at 200°F. and cooled to room temperature before aftertreatment.

The borax buffered dialdehyde starch was prepared by heating 89 grams of tap water and one gram of borax to 60°C. and without further heating, 10 grams of dialdehyde starch was added with stirring. After several minutes, the starch thickened and then went to a fluid dispersion. This dispersion was also at room temperature when used as aftertreatment.

The bisulphite buffered dialdehyde dispersion was prepared by heating 88.5 grams tap water to 70°C., adding 1.5 grams sodium bisulphite, and then adding 10 grams of dialdehyde starch. The temperature was kept between 70° and 75°C. for thirty minutes at which time the dispersion thinned slightly. At room temperature this dispersion was the most viscous. All dispersions were applied with the Bird film applicator which had a clearance of one mil (0.001 inch).

The aftertreated sheets were dried in the oven also and all sheets were conditioned at 73°F. and 50 percent relative humidity before testing.

COATING SERIES II The dialdehyde starch used in series II was prepared with sodium bisulphite as described in the section on aftertreatment. The first coating color in this

series contained: 143 grams clay slip (70 percent solids), 200 grams bisulphite buffered dialdehyde starch (10 percent solids), 4 grams soap (10 percent solids), and 5 grams preservative (10 percent solids). After blending, a very fluid coating color resulted. The viscosity was 44 centipoises at 100 rpm using the number two spindle. The pH was 6.9. The total solids were 30.5 percent and produced a fourteen pound coat weight when a number 24 R.D. wire wound rod was used.

The second coating color in this series was prepared by mixing 450 grams of the previously prepared coating color, in series I, containing 100 percent conventional oxidized starch as adhesive, and 67.5 grams of coating color from this series using 100 percent bisulphite buffered dialdehyde as adhesive. The resultant mixture contained an adhesive ratio of 90 percent conventional starch and 10 percent dialdehyde starch. The total solids content was 44.1 percent, the pH was 6.7 and the viscosity was 665 centipoises at 100 rpm with number three spindle. After centrifuging for foam removal, a number 12 coating rod was used to coat several sheets. The high viscosity of the coating color was a result of remaining in the refrigerator for some time while being stored.

SERIES II AFTERTREATMENT Sheets from each group of coating colors in series II were aftertreated with 10 percent dispersion of conventional oxidized starch and bisulphite buffered dialdehyde starch. The dispersion of conventional oxidized starch was watery and caused the sheets to

wrinkle and form an uneven film; the dialdehyde dispersion was viscous and jelly-like and made a more even film.

CALENDERING AND TESTING All of the coated and after-treated sheets were supercalendered. The pressures on the gauges of the calender were set at twenty pounds per square inch. After conditioning, the sheets were put through the calender four times, once in each direction, with the coated side against the steel roll.

The tests run on the sheets were Dennison wax pick (T 459 m-48), gloss (T 480 m-51), and wet rub resistance. The gloss was evaluated on the Photovolt gloss meter and the sheets were supercalendered on the Wheeler Roll Company laboratory supercalender. The wet rub resistance was evaluated by placing three drops of distilled water on the coated surface about one-quarter inch from the edge of the sheet. At the end of sixty seconds the spot was gently rubbed toward a sheet of black paper. Six such rubs were made on the same spot and the black paper was moved to make six streaks. An arbitrary scale was chosen: poor, poor to fair, fair, fair to good, good, good to excellent, and excellent.

FORMULAE AND EVALUATION
OF
COATING COLORS

	<u>SERIES I</u>						<u>SERIES II</u>					
Experiment Number	1	2	3	4	5	6	7	8	9	10	11	12
<u>PIGMENT</u>												
Coating Clay	1000	1000	1000	1000	1000	1000	1000	1000	1000	1000	1000	1000
Petrasodium pyrophosphate	3	3	3	3	3	3	3	3	3	3	3	3
<u>ADHESIVE</u>												
Conventional oxidized starch	200	200	200	200	180	180	180	180	180	-	-	-
Dialdehyde starch (borax)	-	-	-	-	20	20	-	-	-	-	-	-
Dialdehyde starch(bisulphite)	-	-	-	-	-	-	20	20	20	200	200	200
<u>AFTERTREATMENT</u>												
Conventional oxidized starch	No	Yes	No	No	No	No	No	Yes	No	No	Yes	No
Dialdehyde starch (borax)	No	No	Yes	No	No	Yes	No	No	No	No	No	No
Dialdehyde starch(bisulphite)	No	No	No	Yes	No	No	No	No	Yes	No	No	Yes
<u>EVALUATION</u>												
Dennison wax pick	5.5	9	8.3	9.5	6.5	8.0	7.0	10.0	11.5	10.0	14.0	12.5
Gloss, %	23.6	20.0	18.4	21.0	24.1	18.4	24.0	21.5	24.5	20.7	21.7	20.5
Wet rub resistance	P	P	F	E-G	F-P	F-P	F	E-G	G	F	E-G	E-G

Note: All materials used in coating formulas are expressed in parts by weight. All coating colors contain 0.4 percent (based on pigment) soap, and 0.5 percent sodium pentachlorophenate (as preservative).

CHARACTERISTICS AND APPLICATION DATA
OF
COATING COLORS

	Series I		Series II	
Experiment Number	1 - 4	5 - 6	7 - 9	10 - 12
<u>CHARACTERISTICS</u>				
Total solids %	46.2	47.2	44.1	30.5
Brookfield visc. cps.	380	376	665	44
pH Value	8.8	8.9	6.7	6.9
<u>APPLICATION</u>				
RD rod number	12	13	12	24

All coating colors contained 0.4 percent (based on pigment) soap, and 0.5 percent sodium pentachlorophenate.

DISCUSSION OF RESULTS

DENNISON WAX PICK The addition of 10 percent borax buffered dialdehyde starch increased the wax pick of the control from 5.5 to 6.5. The use of 10 percent bisulphite buffered starch in the control increased the wax pick to 7.0, and the coating color containing 100 percent bisulphite-dialdehyde increased the wax pick to 10.0.

The use of conventional oxidized starch as an after-treatment increased the wax pick of the control 3.5 units,, increased the 10 percent bisulphite by 3.0 units, and the 100 percent bisulphite by 4 units.

Borax buffered dialdehyde as aftertreatment increased the wax pick of the control 3.0 units and the 10 percent borax coating pick by 2.5 units.

Bisulphite buffered starch as aftertreatment produced the most significant increase in the wax pick by increasing the control 4.5 units, the 10 percent bisulphite coating 4.5 units and the 100 percent bisulphite coating 2.5 units.

Bisulphite buffered dialdehyde gave the most significant increase in wax pick when used in the coating as an adhesive or as an aftertreatment.

Borax buffered dialdehyde gave an increase in wax pick when added to the coating as an adhesive, but did not increase the pick strength as much as conventional oxidized starch, when used as an aftertreatment.

The increase in pick strength with aftertreatment was most probably due to the covering of the pigment and coating

with the film of starch and decreasing the penetration of the molten wax into the sheet, as well as introducing more adhesive to the coating.

GLOSS This test was the most insignificant and it was difficult to draw any definite conclusions from the results. The control and 10 percent borax coatings decreased by about 5 percent reflectance after treating with the borax buffered dialdehyde dispersion. The remaining coatings were not significantly changed with or without aftertreatment.

WET RUB RESISTANCE The wet rub resistance increased from poor to fair with the introduction of dialdehyde starch as adhesive. The use of conventional oxidized starch as aftertreatment did not increase the wet rub when used on the control. The application of conventional starch to the other coated sheets increased the wet rub resistance to excellent-to-good.

Borax buffered dialdehyde starch increased the wet rub resistance of the control from poor to fair, but did not increase the resistance of the borax coating.

The application of bisulphite buffered dialdehyde starch as aftertreatment increased the wet rub resistance to excellent-to-good.

The aftertreatment appeared to cover the coating with a film which increased the wet rub resistance. This was true in each case, except the aftertreatment of the control with conventional oxidized starch.

CONCLUSIONS After evaluation of the prepared sheets and examination of the results, the following conclusions were formed:

1. The addition of ten percent borax buffered or bisulphite buffered dialdehyde starch to conventional oxidized starch as adhesive, increases the surface strength of the coating as well as the wet rub resistance.

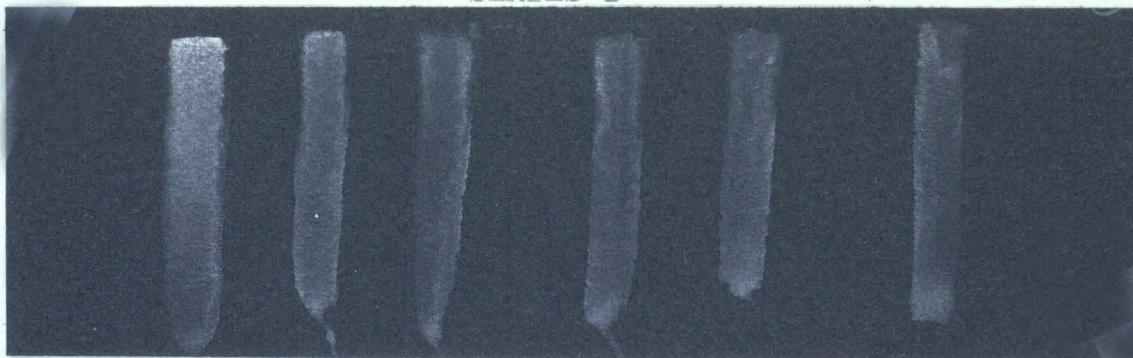
2. Aftertreatment with the starch dispersions increases the surface strength of the coating and the wet rub resistance.

3. Bisulphite buffered dialdehyde starch is a better coating adhesive and "waterproofer" than borax buffered dialdehyde starch.

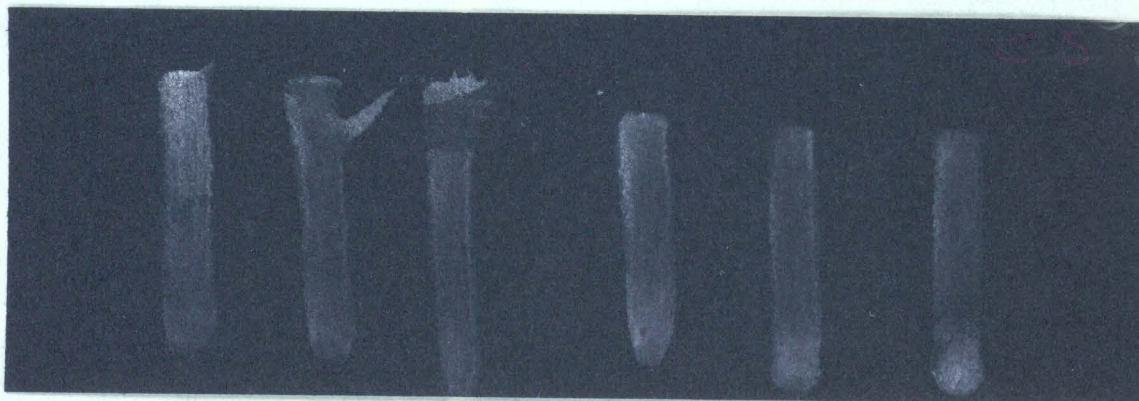
Acknowledgment: I would like to thank Dr. Alfred H. Nadelman and the Department of Paper Technology whose cooperation has made this investigation possible.

EXHIBITS OF WET RUB RESISTANCE EVALUATION

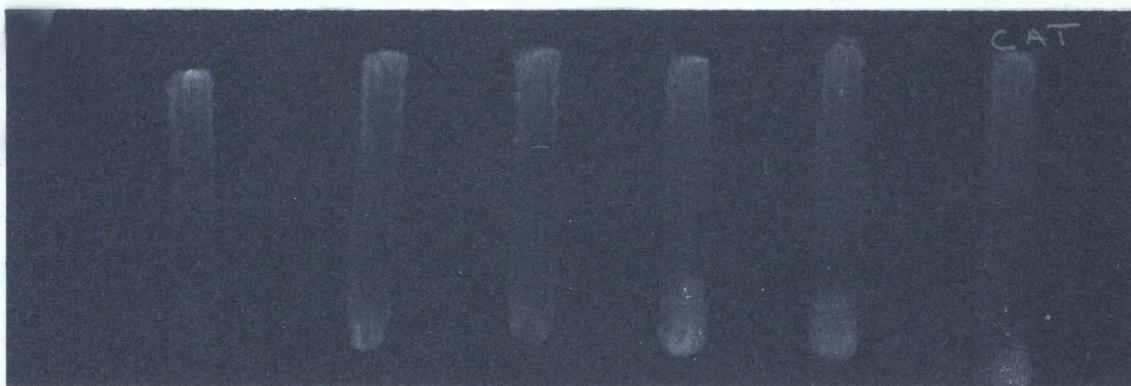
SERIES I



Exp. 1 100 percent Conventional Oxidized Starch

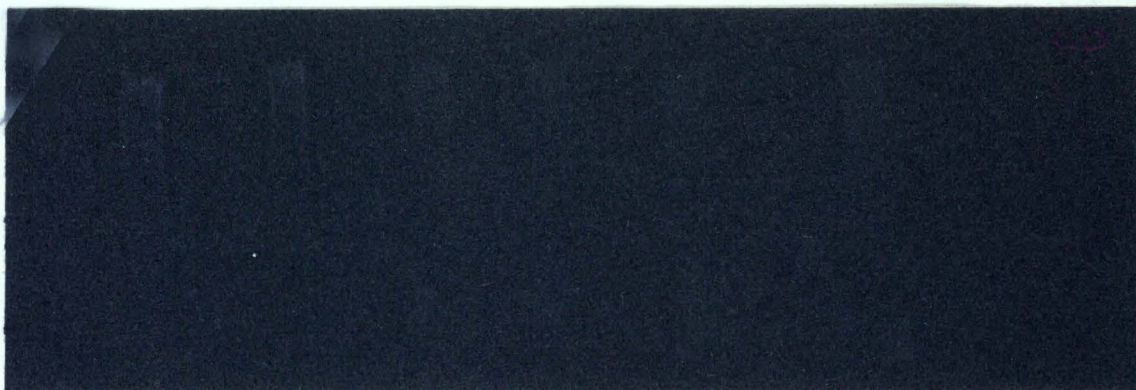


Exp. 2 100 percent Conventional Oxidized Starch
aftertreated with conventional oxidized Starch

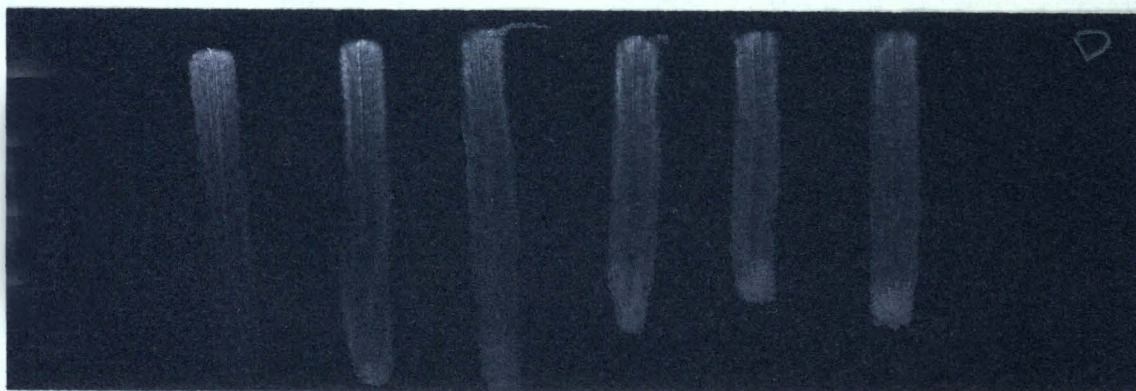


Exp. 3 Conventional Oxidized Starch Aftertreated with
borax buffered dialdehyde starch

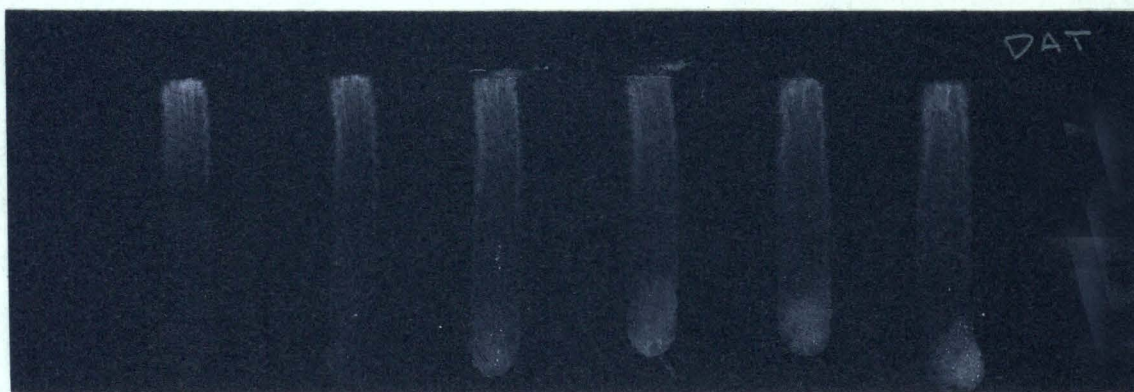
SERIES I



Exp. 4 Conventional oxidized starch aftertreated with
bisulphite buffered dialdehyde starch



Exp. 5 90 percent conventional oxidized starch -
10 percent borax buffered dialdehyde starch

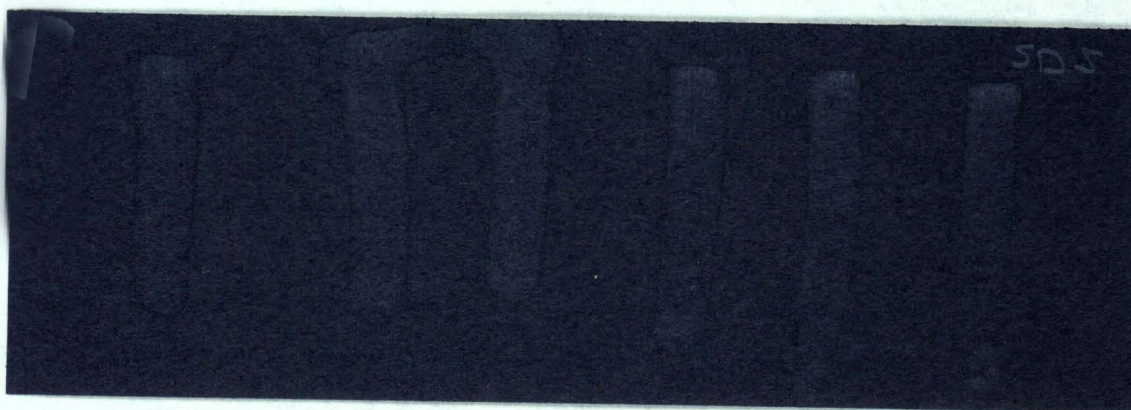


Exp. 6 90 percent conventional oxidized starch -
10 percent borax buffered dialdehyde starch
aftertreated with borax buffered dialdehyde
starch

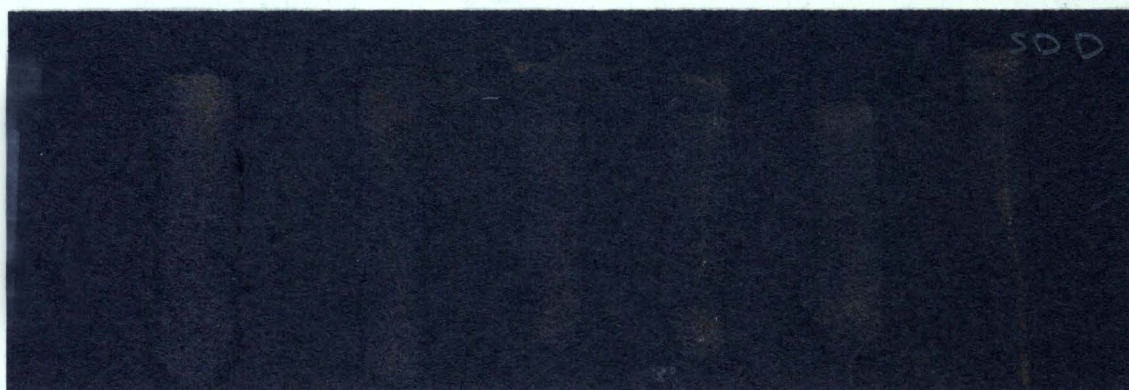
SERIES II



Exp. 7 90 percent conventional oxidized starch -
 10 percent bisulphite buffered dialdehyde starch

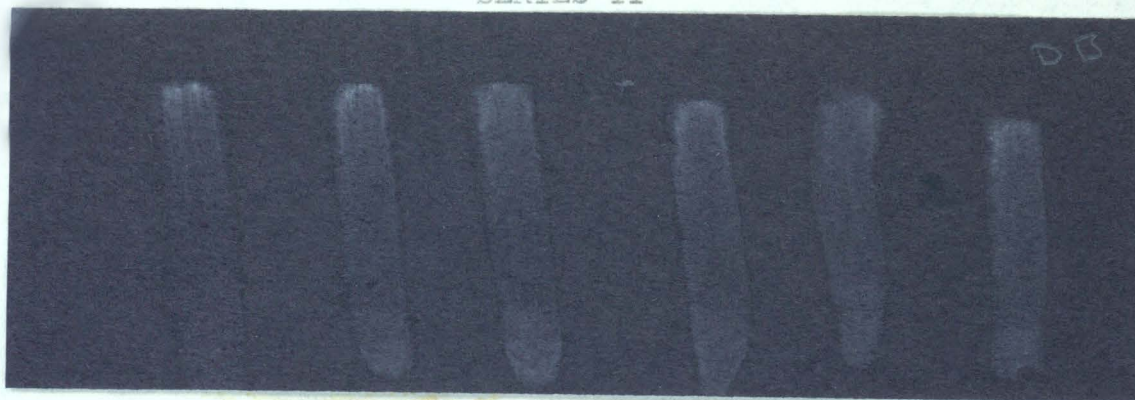


Exp. 8 90 percent conventional oxidized starch -
 10 percent bisulphite buffered dialdehyde starch
 aftertreated with conventional oxidized starch



Exp. 9 90 percent conventional oxidized starch -
 10 percent bisulphite buffered dialdehyde starch
 aftertreated with bisulphite buffered dialdehyde
 starch

SERIES II



Exp. 10 100 percent bisulphite buffered dialdehyde starch



Exp. 11 100 percent bisulphite buffered dialdehyde starch
aftertreated with conventional oxidized starch



Exp. 12 100 percent bisulphite buffered dialdehyde starch
aftertreated with bisulphite buffered dialdehyde
starch